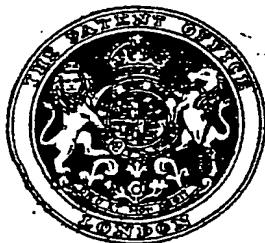


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PATENT SPECIFICATION

737442



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COMPLETE SPECIFICATION

Recovery of Phosgene

We, FARBFENFABRIKEN BAYER AKTIEN-
GESELLSCHAFT, of Leverkusen-Bayerwerk,
Germany, a Body Corporate, organised under
the Laws of Germany, do hereby declare the
invention, for which we pray that a patent
may be granted to us, and the method by
which it is to be performed, to be particu-
larly described in and by the following state-
ment:—

10 This invention relates to a process for the
recovery of phosgene from waste gases con-
taining phosgene and hydrogen chloride.

In the production of isocyanates by treating
amines with phosgene, a large excess of phos-
gene is required. The economy of this process,
therefore, depends on the recovery of substan-
tial quantities of excess phosgene, which is
obtained in admixture with hydrogen chloride
formed in the reaction.

20 It has been proposed to separate phosgene
from hydrogen chloride by washing the waste
gases from this process with cold *o*-dichloro-
benzene. This process is carried out on a large
scale by circulating the *o*-dichlorobenzene,
which is first cooled in a brine cooler, then
passed over a washer where it is brought into
contact with the waste gases containing phos-
gene, heated to 60—70° C. in order to remove
the absorbed phosgene, and then returned to
the cycle. (See BIOS—Miscellaneous Report

30 No. 1; The German Plastics Industry, Appen-
dix 8, pages 6 and 14). This process, however,
has serious drawbacks which cause consider-
able losses of phosgene. Not only are large
quantities of phosgene carried away together
35 with the hydrogen chloride escaping from the
washer, but also the cold *o*-dichlorobenzene,
especially the *o*-dichlorobenzene containing
phosgene, absorbs substantial quantities of
hydrogen chloride, which greatly contaminate
40 the recovered phosgene. Another disadvan-
tage associated with this prior art method is
that the recovered phosgene is in the form of
gas and must be used at once since it cannot
45 be stored as a gas.

The present invention provides a very
simple and economic process for the recovery of
phosgene from waste gases containing phos-

gene. Other advantages of the process are set
out below.

In accordance with the present invention,
we have found that phosgene can be recovered
from waste gases containing phosgene and
hydrogen chloride by passing the waste gases
upwardly through a cooling apparatus which
cools the gases to a temperature below the
boiling point of phosgene and above the boil-
ing point of hydrogen chloride, down which
cooling apparatus liquid phosgene condensed
from the waste gases by the cooling trickles
under gravity.

Preferably the gases are cooled in the cool-
ing apparatus to a temperature of —40 to
—60° C. The gas mixture of hydrogen
chloride and phosgene entering the cooling
apparatus is preferably at a temperature below
the boiling point of phosgene (8° C.), i.e. 0 to
5° C., and is further cooled by contact with
the condensed liquid phosgene which trickles
down the cooling apparatus under gravity.
Liquid phosgene issues through the bottom of
the apparatus and gaseous hydrogen chloride
escapes at the top. The liquid phosgene
obtained by the herein-described recovery pro-
cess contains up to 0.7% of hydrogen chloride
and the escaping gaseous hydrogen chloride
still contains some phosgene, the quantity
depending upon the partial pressure at the
temperature of the escaping residual gas.

The gas mixture of hydrogen chloride and
phosgene is preferably cooled in a tubular
cooler, the tubular parts of which are cooled
by an appropriate cooling medium, for
instance boiling difluorodichloromethane or
difluoromonochloromethane.

The invention is further illustrated by way
of example in the accompanying diagrammatic
drawings, which show sectional views of two
apparatus wherein phosgene can be recovered
from waste gases containing phosgene by the
herein-described process.

Referring first to Fig. 1, the waste gases
enter the cooler 2 from below through the
pipe 1 and pass upwardly through the cooler
where they come into contact with condensed
liquid phosgene which trickles downwards

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under gravity and discharges into the storage tank 4. The non-condensed gases escaping from the cooler 2 through the pipe 5 consist substantially of hydrogen chloride which is 5 eliminated in the washer 6 by irrigation with water fed through the pipe 8 and discharged as an aqueous solution through the pipe 7. Any inert gases which may be present are exhausted through the pipe 9 by the suction 10 fan 10, which is arranged at the end of the apparatus and maintains a slight sub-atmospheric pressure within the whole apparatus, and forced into a chimney. The phosgene, which is obtained in the liquid state, can be 15 taken from the storage tank 4 whenever it is desired.

The amount of phosgene which is recovered by the herein-described process is substantially larger than that obtained according to 20 the above-mentioned prior art process. It is a surprising feature of the present invention that the recovered phosgene contains only 0.5—0.7% of hydrogen chloride, which allows the phosgene to be used for reactions with amines 25 without purification.

In the process for the recovery of phosgene just described, difficulties may arise when the apparatus is leaky. Moisture will then be sucked into the apparatus together with air 30 and, in admixture with the hydrogen chloride, will cause strong corrosion. Not only may such corrosion endanger the operation of the apparatus, but the recovered phosgene is contaminated with metal chlorides, especially iron 35 chloride, which limit its usefulness since metal chlorides cause inconveniences when the recovered phosgene is again used to treat amines.

It is within the scope of the invention to 40 obviate these difficulties by maintaining a slight superatmospheric pressure in the cooler. This is accomplished, as shown in Fig. 2, by arranging a preferably automatic, high pressure relief valve 11 behind the cooler 2 into 45 which the waste gases are led through the pipe 1 and from which the liquid phosgene flows into the tank 4. A pressure of 100—200 mm of water is maintained in the cooler by means of this high-pressure relief valve 11. By this 50 arrangement any moisture is safely prevented from entering the cooler and the recovered phosgene is water-clear. Any leakages are

immediately perceived due to the phosgene odour, and can be remedied. The residual gases, as in the case of Fig. 1, are passed 55 through the pipe 5 into the washer 6 where the hydrogen chloride is eliminated with water admitted through the pipe 8. The hydrogen chloride in the form of an aqueous solution, is discharged through the pipe 7. The washer 6 60 is connected with the suction fan 10 by the pipe 9. The washer 6 connected at the outlet side of the recovery apparatus is preferably maintained at the sub-atmospheric pressure of the suction fan 10 arranged at the end of the 65 recovery apparatus. In this way a sub-atmospheric pressure may be temporarily produced and maintained in the recovery apparatus by means of a by-pass pipe 12 which by-passes the high pressure relief valve 11, should a sub-atmospheric pressure be required for any reason.

What we claim is:—

1. Process for the recovery of phosgene from waste gases containing phosgene and hydrogen 75 chloride, which comprises passing the waste gases upwardly through a cooling apparatus which cools the gases to a temperature below the boiling point of phosgene and above the boiling point of hydrogen chloride, down 80 which cooling apparatus liquid phosgene condensed from the waste gases by the cooling trickles under gravity.

2. Process as claimed in claim 1, wherein the cooling is carried out at a pressure slightly 85 above atmospheric.

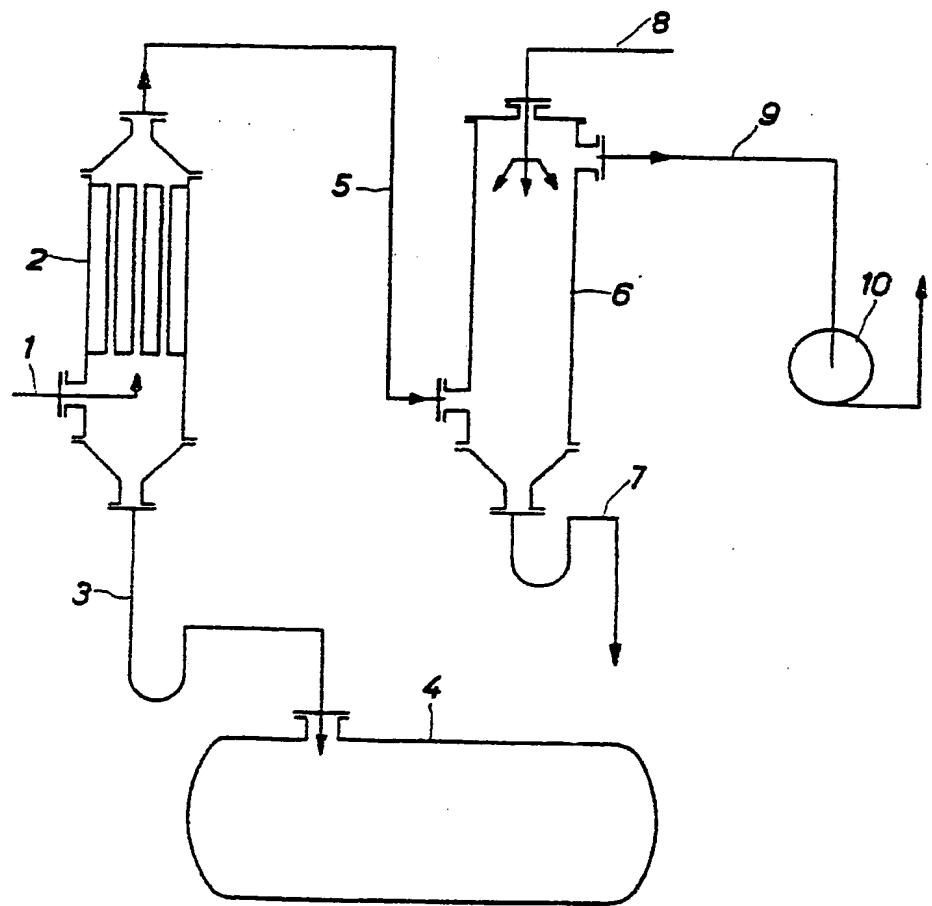
3. Process as claimed in claim 1 or 2, wherein the waste gases are cooled in the apparatus to —40 to —60° C.

4. Process as claimed in any of claims 1 to 90 3, wherein the waste gases entering the cooling apparatus are at a temperature of 0 to 5° C.

5. Process for the recovery of phosgene from waste gases containing phosgene and hydrogen 95 chloride substantially as described with reference to Fig. 1 or Fig. 2 of the accompanying drawings.

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FIG.1



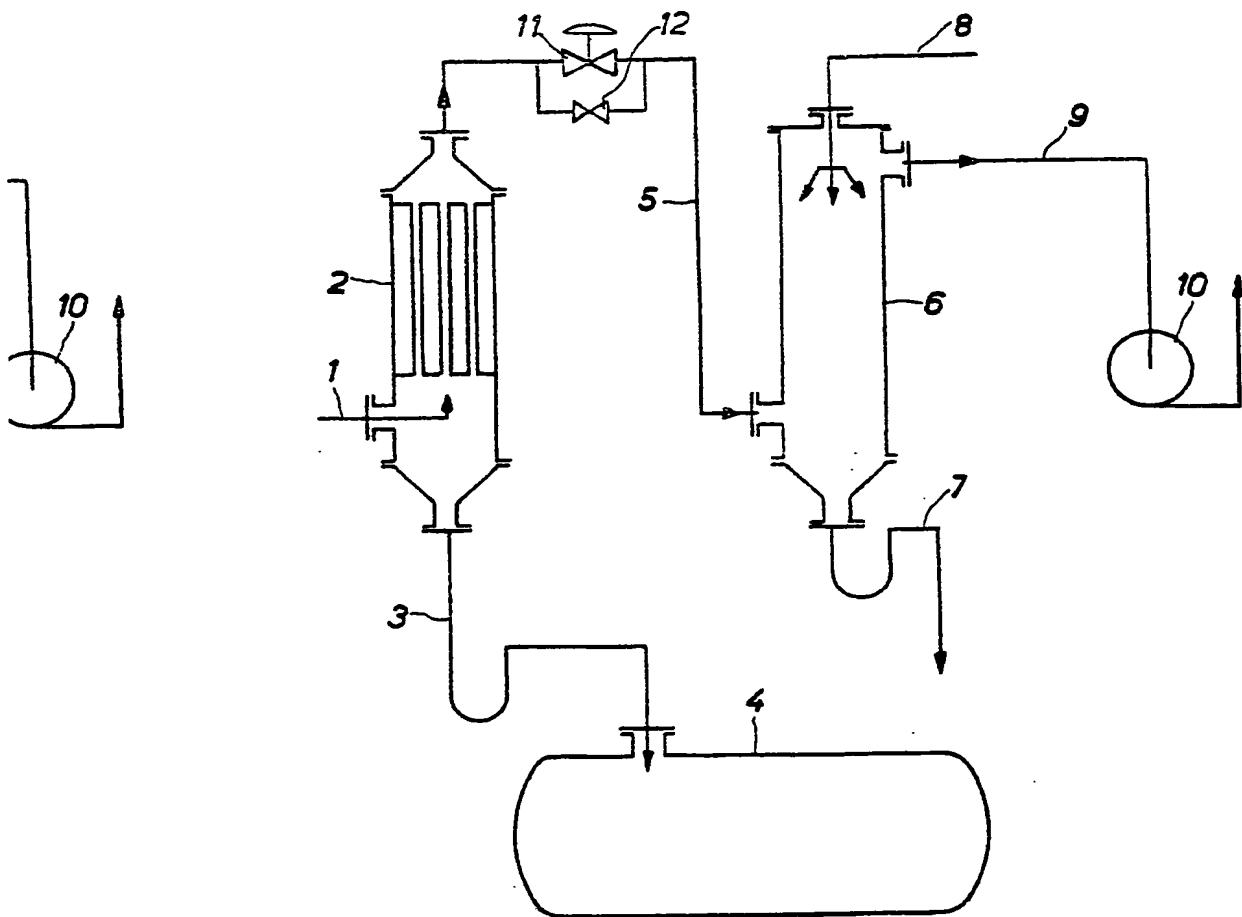
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COMPLETE SPECIFICATION

2 SHEETS

This drawing is a reproduction of
the Original on a reduced scale.
SHEETS 1 & 2

FIG.2



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SHEETS 1 & 2

FIG.1

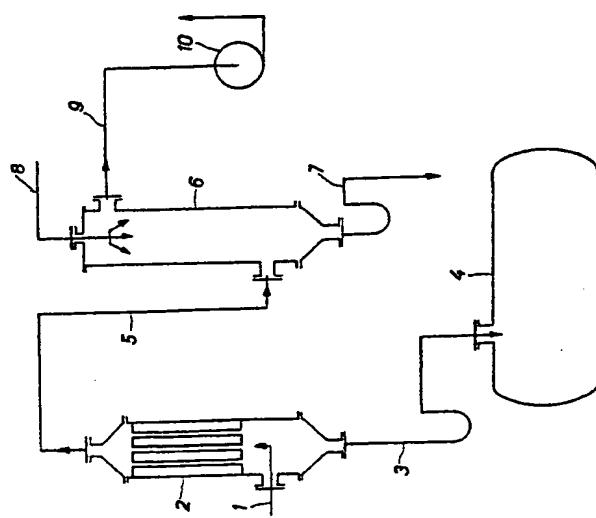
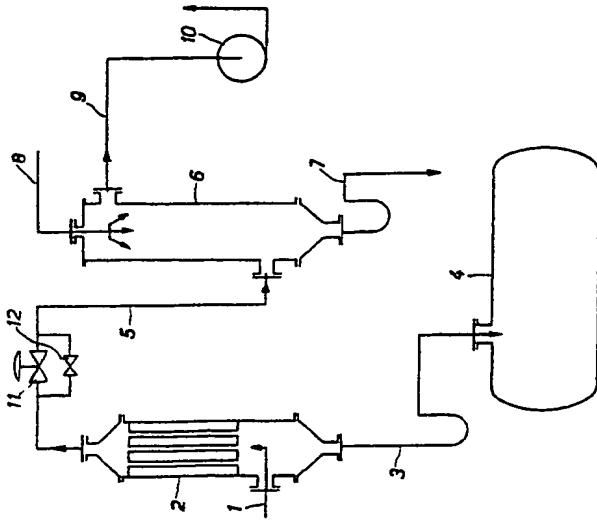


FIG.2



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